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# Improvements in apparatus for observing objects disposed in fluid-tight chambers


**Patent number:** GB971141
**Publication date:** 1964-09-30
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**Classification:**
**- international:**
**- european:** B25J21/02, G02B21/00
**Application number:** GB19630004296 19630201
**Priority number(s):** FR19620886834 19620202

**Also published as:**
LU43092 (A)
FR1323552 (A
BE627235 (A



Abstract of **GB971141**

971,141. Glove boxes. COMMISSARIAT A L'ENERGIE ATOMIQUE. Feb. 1, 1963 [Feb. 2, 1962], No. 4296/63. Heading B4Q. [Also in Division G2] A microscope is mounted outside a glove box 19, but with its objective passing through a flexible seal 28, 29 into the box which contains an evacuated heated chamber 2 containing a specimen 1, e.g. a radio-active metal. The microscope includes a rotary illuminating device 36, polarizers 34, 35 and an objective 24, and is supported on a plate 22 which can be moved in two perpendicular directions, e.g. by screw 30. The second chamber 2 can be heated by the heating coil 14 and reflectors 15, evacuated by the vacuum pump 8 and, if necessary, the liquid nitrogen trap 9, and contains jaws 13 which hold the specimen and a quartz window 6 for the passage of light rays. Circuit diagrams are given for varying the voltage across the resistor 14 to give a variable-temperature heat- ing furnace.


FIG.1.

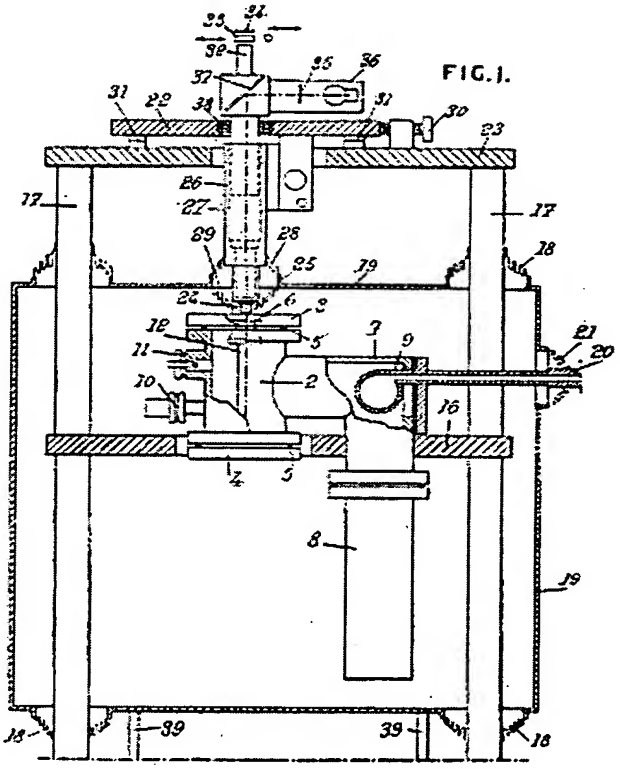
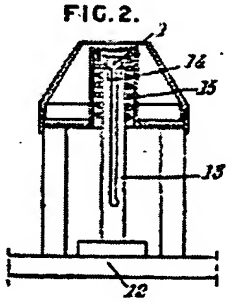


FIG.2.



# Improvements in apparatus for observing objects disposed in fluid-tight chambers

Description of **GB971141**

PATENT SPECIFICATION

971,1 Date of Application and filing Complete r<Xi( \Specification: February 1, 1963 No 429 t

Application made in France (No 886,834) on February 2, 1962.

Complete Specification Published: September 30, 1964.

age < (D Crown Copyright 1964.

Index at Acceptance:-G 2 J ( 8 B, 8 C, 8 M 5, 8 X, 30); B 4 Q 2.

International Classification:-G 02 d (G 21).

The inventors of this invention in the sense of being the actual devisers thereof within the meaning of Section 16 of the Patents Act 1949 are Jean Marie Chevallier and Pierre Rousseau, both French citizens of 39 Rue Mademoiselle, Paris, (Seine), France, and 3, Rue Vauquelin, Paris (Seine), France, respectively.

COMPLETE SPECIFICATION

DRAWINGS ATTACHED Improvements in Apparatus for Observing Objects Disposed in Fluid-Tight Chambers V We, COMMISSARIAT A LUENERGIE ATOMJ Qu E, an organisation created in France by Ordonnance No 45-2563 of October 18th, 1945, of 69, Rue de Varenne, Paris, (Seine), France, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

This invention relates to apparatus for observing, with the aid of a microscope, objects disposed in an evacuated heated chamber notably highly oxidisable metals and alloys and more especially specimens of radioactive metals, which are to be observed at various temperatures, so that it is necessary to work in vacuo by reason of the oxidisability of the metals.

The invention has for its object to obviate the difficulties hitherto encountered in known constructions in mounting a heated and exhausted chamber on a microscope stage.

it is first of all to be noted that since radioactive metals are generally highly oxidisable it is necessary to effect heating in tacuo and at minimum residual pressure.

On the other hand, since the oxides which are likely to be formed are frequently finely powdered, it is necessary to work in a glove box in order to ensure protection of the operator against radioactive dust.

Having regard to these special conditions, therefore, the microscope would normally be mounted in the glove box and the exhausted heating chamber would be mounted on the stage or stand of the microscope which would give rise to considerably difficulties in view of the necessity 4 to render the stage movable, and therefore also the said exhausted chamber, in relation to the objective in order to permit exploration of the field.

According to the present invention there is provided apparatus for observing, objects 45 submitted to heat in an evacuated, heated chamber having an observation window, wherein the heated chamber is disposed inside a glove box and a microscope is mounted outside the glove box but with 50 its objective passing into the box through a flexible seal, the arrangement being such that the microscope may be manipulated entirely from outside the glove box.

The microscope may be mounted on a 55 fixed support in such manner as to be movable as required on the support outside the glove box (in order to permit transverse displacements of the exploring beam).

In addition, if it is desired to be able to 60 operate with polarised light, the microscope may be provided

with a source of illumination adapted to coact with an appropriate Nicol prism or polariser, again outside the glove box, so that all the opera 65 tions on the microscope take place outside the said glove box.

The invention will be further described by way of example with reference to the accompanying drawings, in which: 76 O Figure 1 shows one embodiment in elevation, and partly in section, Figure 2 shows, in vertical section, a furnace for heating a specimen, Figures 3 and 4 are two circuit diagrams 75 relating to regulating means for the furnace, and Figure 5 is a curve of temperature versus time.

The fluid-tight furnace intended to receive 80 r.

4 T 1/63 971,141 the specimen to be examined (specimen 1, Figure 2), consists of a cylindrical body 2 sealed at both ends by end plates 3 and 4 with packing rings 5 One of the end plates 3 is formed with an aperture which is closed by a plane quartz plate 6 having parallel faces for the microscopic examination The plate is bonded or fused to the end plate.

The chamber 2 is connected by a duct 7 to a motor-pump set 8 adapted to produce an ultra-high vacuum In addition, there is shown at 9 a liquid-nitrogen trap.

An ionisation gauge 10 is provided for measuring pressure and an auxiliary crucible 11 is for use in certain operations More especially, if it is desired to reveal the structure of the metal undergoing the experiment, the auxiliary crucible may be employed to subject it to the action of a gas or vapour obtained by volatilising a salt The chamber 2 is advantageously double-walled and cooled by water circulation.

The specimen 1 to be examined is disposed within the fluid-tight furnace, being mounted, for example, as illustrated in Figure 2, on a support 12 terminated by jaws 13 which grip the specimen at their ends This securing means ensures reproducible orientation of the specimen in relation to the optical axis of the system, which orientation is maintained during heating It also enables the specimen to expand freely without stress.

The heating source for bringing the said specimen to the desired temperatures, comprises a coiled heating resistor 14, which is introduced into the interior of reflecting metal screens 15 The heating resistor preferably consists of a nickel-chromium conductor with an insulating sheathing of powered magnesia and an external sheathing consisting of a metal such as Iconel (Trade Mark).

In the case of high temperatures, it is also possible, for the purpose of avoiding any parasitic metallisation of the specimen, to employ a molybdenum resistance coil, :5 ( since the metal retains very good mechanical stability at elevated temperature The coil could be fixed on an alumina rod by means of platinum wires.

It is also to be noted that the metal of the specimen 1 is preferably insulated from contact with the jaws on the member 13 by a very thin foil of appropriate metal.

The whole fluid-tight chamber 2 is mounted on a support 16 secured to Gio columns 7 extending by way of bellows 18 through a glove box 19 surrounding the chamber and its associated elements.

A duct 20 extends to the trap 9 through sealing bellows 21.

The microscope for observation through the quartz window 6, is supported by a plate 22 mounted on a table 23 outside the glove box 19, and its objective 24 can be positioned opposite the window 6 as it passes through an appropriate aperture 25 70 in the wall of the box, sealing bellows again being used to maintain fluid-tightness of the glove box.

In the illustrated construction, the lensholder body or tube 26 of the microscope 75 is connected to the plate 22 by a micrometer device 27, enabling vertical adjustment and tile fluid-tightness with the glove box to be obtained by means of two bellows 28 and 29 which connect the body 26 and 80 the tube of the objective 24 to the edge of the aperture 25.

In order that the field of view may be shifted so that the whole of one surface of the specimen may thus be explored, 85 means are provided for displacing the plate 22 These means comprise a screw 30, the plate resting on the table 23 through studs 31, of which there may be three A further screw, on a line normal to that of the screws 9 G 30, may be provided for adjustmenting the plate in two perpendicular directions.

In addition, especially when it is desired to operate under polarising light, a rotatable illuminator may be provided in com 95 bination with one or more polarisers or Nicol prisms In the illustrated construction, there is shown at 32 the eyepiece, at 34 a polariser and at 33 a polarisation interferometer The polariser

34 coacts with the 100 polariser 35 provided in the illuminator 36, acting with a prism 37, the whole being so mounted as to be rotatable about the axis of the lens-holder tube, with ball bearings 38, the lens-holder tube 26 and 105 objective not rotating.

It will be seen that with such an assembly the handling of the microscope takes place entirely outside the glove box, with the possibility of utilising any of the usual 110 observation techniques, more especially by rotation of the illuminator and illumination with polarised light In addition, such handling does not in any way detrimentally affect the conditions of fluid-tightness of 115 the glove box 19 and of the fluid-tight chamber 2.

It is to be noted that, before the apparatus is employed, the heating chamber 2, its furnace 14, 15 and the trap 9 must be de 120 gassed For this purpose, detachable heating elements, not shown in the drawings, will be employed.

The columns 17 rest on the ground The fact that they are not directly connected to 125 the glove box, but are connected thereto by way of bellows 18 or other flexible intermediate sealing means, makes it possible, by avoiding any rigid connection, to prevent transmission of vibrations which 130 971,141 might detrimentally affect the reproduction of a photomicrograph of the specimen The g love box in turn rests on legs shown at 39.

The heating devices may differ from tnose referred to For example, induction heating, or heating by electron bombardment could be employed.

There will advantageously be provided in io combination with the assembly an automnatic regulating system for maintaining the temperature at desired and adjustable values, the said system comprising thermocouples adapted to co-operate with an auto-j 5 matic control system.

More especially, it may be desirable to obtain a temperature rise in accordance with a substantially linear curve representing the temperature T as a fujiction of time 2 ò t, such as the curve C in Figure 5, so as to show the singular points or levels such as P corresponding to a phase change of the metals or alloys to be examined.

For this purpose, two voltages may be set up, in opposition, namely a first or control voltage, for example emanating from a rheostat of which the slider effects substantially linear variation of the said voltage, while the other is extracted from a thermocouple subjected to the temperature of the furnace, which is electrically heated by a heating circuit controlled by a second rheostat, the whole being combined with driving means controlled by the error voltage emanating from the comparison of the aforesaid two voltages and acting on the second rheostat in order to ensure the desired heating temperature at each instant.

The driving means may be set in operation in one direction or the other with the aid of a relay influenced by the error voltage, so as to actuate the second rheostat in a succession of steps.

In a first constructional example illustrated in Figure 3, the aforesaid arrangement is operated in the following manner.

The first rheostat 41 is fed by a source 42 and has its slider driven by a motor M 1, for example of the synchronous type, of which the speed is chosen in accordance with the desired temperature rise curve.

The voltage across the terminals of the rheostat at ac is therefore the aforesaid control voltage.

This voltage is applied at b in opposition to that produced by a thermocouple subjected to the furnace temperature The thermocouple may be soldered to the heating resistance 14 of the furnace.

jo The error voltage set up in the circuit abc then acts on a sensitive electronic or other relay 46 and, depending upon its direction, it opens or closes an independent circuit 47 comprising one or more reversi S ing relays, of which the coil is shown at 48.

In the manner which will hereinafter be indicated, the latter relay starts in one direction or the other the driving system intended to control the slider of the second rheostat, shown at 49, which is adapted to 70 vary the voltage of the heating circuit 14, which circuit receives its feed, for example from the mains, at S.

The illustrated driving system comprises a motor M 2 having two windings illustrated 75 at 50 and 50 ', for producing the rotation in the two respective directions, the said motor being intended to actuate the slider of the rheostat 49 and being controlled by a switch I from the relay 48, while also 8 g being dependent upon circuit-breaking contacts influenced by the motors which will hereinafter be referred to.

Two other motors ml and n, controlled by a second switch i from the relay control 85 through cams C 1, C 2 sets of contacts 52, 53 and 52 ', 53 ', the first motor ml becoming operative when the motor Ml is rotating in one direction (winding 50), while the second motor in_ becomes operative for the 90 other direction (winding 50 ').

There are two cams Cl which act simultaneously so as to open a contact 52, while the other 53 is closed The same is the case with two cams C, in relation to 95 the contacts 52 ', 53 ' The cams in each pair are appropriately positioned.

The driving system operates as follows.

The reversing relay I, i being, for example, first of all in the position 0, 1, 100 as illustrated in Figure 3, the following actions occur as the cams C 1 travel past their microcontacts 52, 53:

The link shown at ABC opens at 52, but the motor inm, which was rotating, con 105 tinues to rotate because it is fed by the link AC:

The link EFG closes at 53, so that the motor M 2 is fed through its winding 50 and drives, during the time when the con 110 tact 53 is closed, the movable arm of the rheostat 49.

The same actions occur at each revolution of the motor ml, and this results in a variation by pulses, i e in steps, of the 115 voltage across the terminals of the furnace at 14 This continues until the instant when the current changes its direction in the circuit abc and the switches I, i take up the position 0, 2 120 At this instant, the motor inm stops and it is then the motor m 2 which starts and, in combination with the winding 50 ' of the motor M 2 and with the contacts 52, 52 ' and 53, 53 ', gives rise to a similar operation 123 It is to be noted that the motor ml stops at the passage of the cam C 1, which opens the link Al BC and closes the link EFG In this position, the whole arrangement is ready to transmit a pulse immediately the 130 971,141 reversing relay brings the contacts I, i back to 0, 1 There is therefore no dead time, and this is one of the advantages of the multi-motor arrangement.

Finally, at equilibrium, the arm of the rheostat 49 undergoes a reciprocating movement about a position of equilibrium Any tendency of the furnace temperature to fall or to rise therefore has the effect of modifying the period of this movement.

In an arrangement of this type, there will be, for example, two rates of variation of the temperature, one of 100 C per hour and the other of 2000 C per hour The speed of rotation of the motors driving the cams will be, for example, one revolution in 6 seconds The cams will have a useful angle of about 150, these particulars being given by way of example only.

Recordings of the temperature as a function of time, obtained with the abovedescribed type of furnace, show a temperature fluctuation of the order of a degree, the drift of the curve being zero.

In the arrangement of Figure 3, in which the regulating action is exerted without time lag, it is possible to obtain a circuitbreaking period of the order of 30 s This period is sufficient to permit a slight time lag in the regulating action, which simplifies the construction of the arrangement, for example in accordance with the following embodiment.

In this embodiment, in which the two windings of the motor M actuating the rheostat 49 have again been denoted by 50 and 50 ', the successive oneration of the two windings in controlled with the aid of a single cam C, which acts on two contacts 53, 53 ', this cam being continuously driven bv a motor in.

Depending upon the position of the reversing relay, the corresponding winding is energised throughout the period when the cam is passing In the most unfavourable case, the regulating action is exerted with a time lag equal to the time taken by the motor driving the cam to perform one revolution (that is the case where the reversing relay changes its position at the instant when the cam ceases to act on the corresponding switch).

Although less accurate, this system may be suitable in many cases.

# Improvements in apparatus for observing objects disposed in fluid-tight chambers

Claims of **GB971141**

WHAT WE CLAIM IS:-

1 Apparatus for observing objects submitted to heat in an evacuated, heated chamber having an observation window, wherein the heated chamber is disposed inside a glove box and a microscope is mounted outside the glove box but with its objective passing into the box through a flexible seal, the arrangement being such that the microscope may be manipulated entirely from outside the glove box.

2 Apparatus according to claim 1.

wherein the microscope is mounted on columns which pass through the glove box by way of flexible seals, the heated chamber being supported inside the box by the same 70 ' columns.

3 Apparatus according to claim 1 or 2, wherein the heated chamber for containing the specimen consists of a cylindrical or other body sealed at both ends and hav 75 ing at one end a quartz window for the passage of the light rays.

4 Apparatus according to claim 1, 2 or 3 comprising jaws within the heated chamber for holding the specimen to be 80 examined.

Apparatus according to claim 1, 2.

3 & 4, where the heated chamber contains an electric resistor inside a reflector for heating the specimen 85 6 Apparatus according to any of claims 1 to 5 wherein the heated chamber is connected to an ultra-high vacuum pump whicii comprises in addition, if necessary, a liquidnitrogen trap 90 7 Apparatus according to any of claims 1 to 6, wherein the microscope is suppor Aed by a plate adapted to be displaced transversely in relation to the axis of the microscope to enable the specim-en to be 95 examined.

8 Apparatus according to any of claims 1 to 7, w-nerein the objective of the microscope passes through bellows into the glove box 10 9 Apparatus according to any of claims 1 to 8, wherein the microscope comprises a rotatable illuminator.

Installation according to claim 9, wheiein the illuminator is combined with 10 polarisers to provide illumination with polarised light.

11 Apparatus according to any of claims 1 to 10, wherein the chamber containing the specimen comprises a variable 11 temperature heating furnace, provided with a regulating system in which there are set up in opposition to one another two voltages, namely a first control voltage arranged to vary with time in accordance 11 with a predetermined law, the other voltage being derived from a temperature sensitive device subjected to the temperature of the furnace, which is electrically heated by a heating circuit controlled by a rheostat 12 which is driven by driving means controlled by an error voltage emanating from the comparison of the said two voltages.

12 Apparatus according to claim 11, wherein the control voltage is provided by 12 a rheostat whose slider is driven at the required rate.

13 Apparatus according to claim 11 or 12, wherein the control voltage varies linearly 13 971 141 14 Apparatus according to claim 11, 12 or 13, wherein the driving means comprise a motor operated in one direction or the other with the aid of a relay influenced by the error voltage and arranged to adjust the rheostat by successive steps.

Apparatus according to claim 14, wherein the successive steps are produced by two auxiliary motors which operate respectively when the main motor rotates in one direction and the other, and act on cams controlling switches for the main motor, the whole being so arranged as to avoid dead times.

i 16 Apparatus according to claim 14, wherein successive pulses are provided by a single constantly rotating auxiliary motor which drives at least one cam which acts on switches provided in the winding

circuits of the main motor 20 17 Apparatus substantially as hereinbefore described with reference to and as shown in Figs 1 and 2 of the accompany drawings.

18 Apparatus substantially as herein 25 before described with reference to and as shown in Figs 1 and 2 in combination with Fig 3 or Fig 4 of the accompany drawings.

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Berwick-upon-Tweed: Printed for Her Majesty's S tationery Office by The Tweeddale Press Ltd -1964 Pu'l'ished at The Patent Office 25 Southampton Buildings, London, W C 2 from which copies may be obtained.

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